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# TITANIUM-GETTER TESTING FOR TMX-U

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# TITANIUM-GETTER TESTING FOR TMX-U*

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## ABSTRACT

This report summarizes the results of titanium-gettering tests performed during August and September of 1983. Several current and voltage schedules were evaluated for possible implementation with TMX-U's computer-controlled getter system. The tests were performed using 1/8-inch wire composed of 85% Ti and 15% Ta. Measurements of wire temperature as a function of getter current were made. We performed glow discharge cleaning (GDC) of the vessel with wires in place to determine if GDC had any adverse effect on wire lifetime.

## INTRODUCTION

Titanium gettering is used extensively in the Tandem Mirror Experiment-Upgrade (TMX-U) to condition walls, to reduce impurity levels, and to pump excess cold deuterium gas. During the experimental cycle that ended in August 1983, there was a high failure rate for getter wires.[1] These failures prompted a series of experiments in a 3100-liter test vessel to determine if the failures could be related to a material property of the wire or to some aspect of machine operation such as glow discharge cleaning (GDC).

The getter system on TMX-U is being automated, and we expanded the scope of these getter experiments to support this transition to computer-controlled power supplies. Software developed for the HP-9000-based control system was tested along with the hardware modifications necessary to remotely control the TMX-U getter power supplies. The resistance of getter wires changes as titanium is evaporated from their surface. As a result, it is necessary to operate getters on a current schedule in order to maintain fairly even deposition throughout the lifetime of the wire.[2] Several schedules were tested, and the schedule shown in Fig. 8 was selected for the initial tests of the automated system on TMX-U.

## EXPERIMENTAL EQUIPMENT

We performed these tests in the 3100-liter tank in Room 1167 of Building 435. Figure 1 is a photograph of the vessel showing the port through which an Inficon Model XTM Film Thickness Monitor (XTM) was inserted. The XTM measured the thickness of titanium deposited on the surface of a crystal approximately 20 inches from the getter wires. Aluminum disks like the one shown in Fig. 2 were used to calibrate the XTM. The XTM provided very useful data, but was too temperature-sensitive to permit measurements during the getter cycle, even though we used water cooling. When the getters came on, the crystal was heated. The monitor interpreted the accompanying increase in crystal vibration frequency as a drop in thickness. About 40 seconds after the getters shut off, the crystal cooled and the output stabilized. Two of these disks were sent to Sandia National Laboratories for analysis; the results of this analysis[3] are shown in Table 1. The thickness was measured by Rutherford backscattering (RBS) and the composition was determined by proton induced x-ray emission (PIXE). The XTM agreed to within about 10% of the RBS measurements. The mercury found in the samples came from the Westinghouse 9-inch mercury diffusion pump on the tank.

The first 12 wires were run in two batches of six 70-inch wires; these wires were connected to a "pole getter" similar to the one in Fig. 3. We ran a total of 17 wires (66 inches long) on a "harp getter" like the one in Fig. 4.

## RESULTS OF EXPERIMENTS

The first set of six wires was run using a constant voltage schedule similar to the method used in the past on TMX-U. We first heated the wires for 30 minutes at 60 amperes and then adjusted the power-supply voltage to obtain 106 Amps of current in the wire. This voltage was held constant for 200 cycles. One-minute-on, one-minute-off cycles were controlled by a power

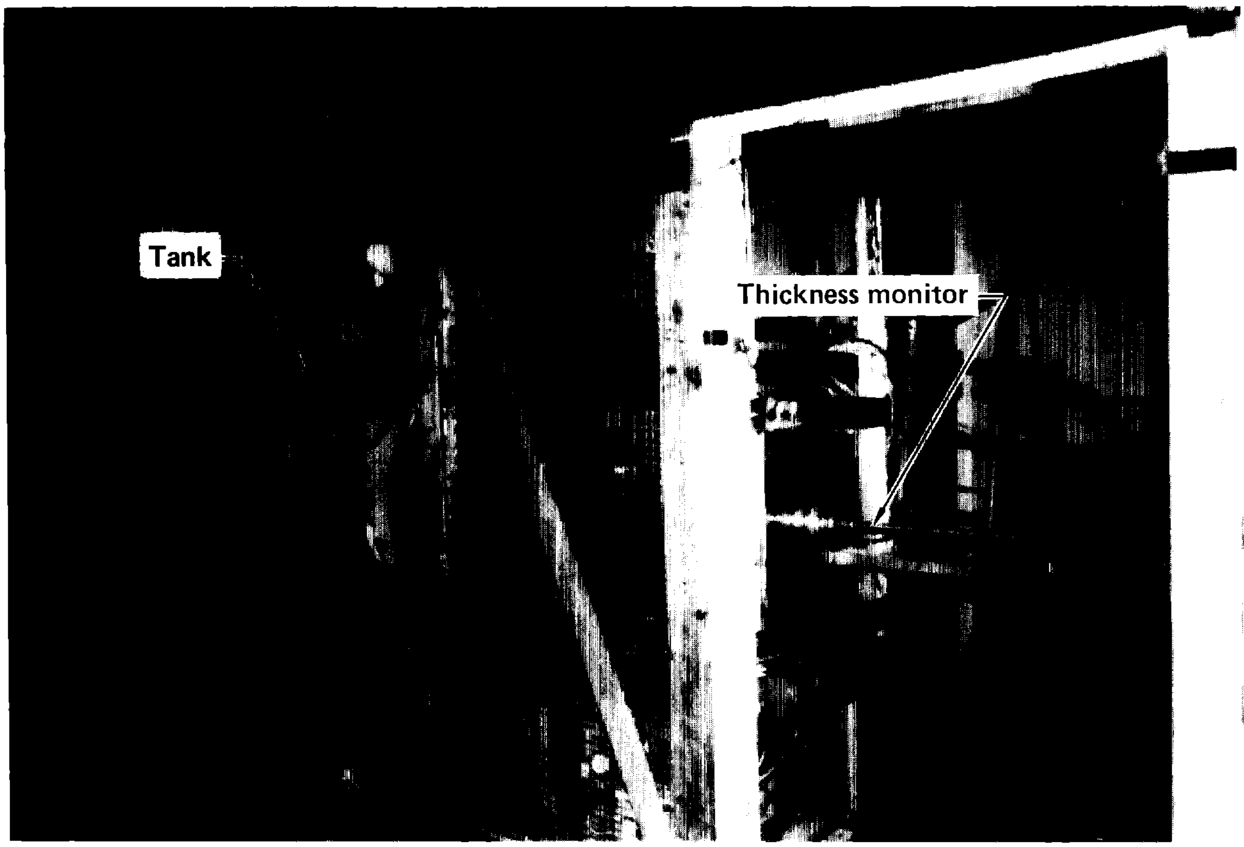



Fig. 1. Test vessel and thickness monitor.

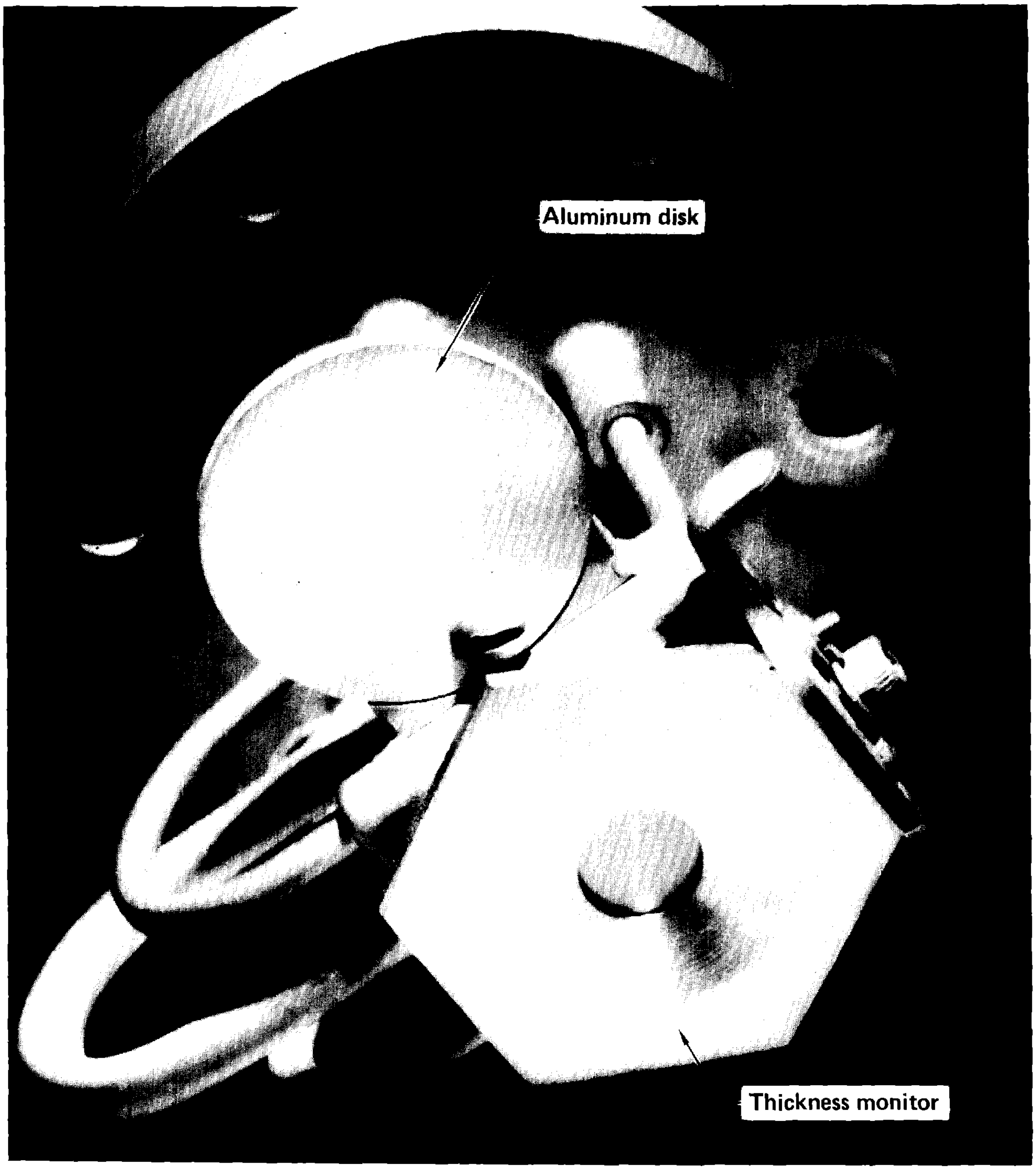



Fig. 2. Thickness monitor crystal and aluminum disk used to calibrate the XTM.

Table 1. Film-thickness and composition data for two samples.

| Sample | XTM[a] measured thickness (Å) | Sandia (RBS[b]) measured thickness (Å) | Sandia (PIXE[a]) measured composition %Ti | %Ta | %Hg |
|---|---|---|---|---|---|
| 1 | 34,900 | 32,800 | 98 | 0.1 | 2 |
| 2 | 37,200 | 41,500 | 99.7 | trace | 0.3 |

[a]Inficon Model XTM Film Thickness Monitor.

[b]Rutherford backscattering.

[c]Proton induced x-ray emission.

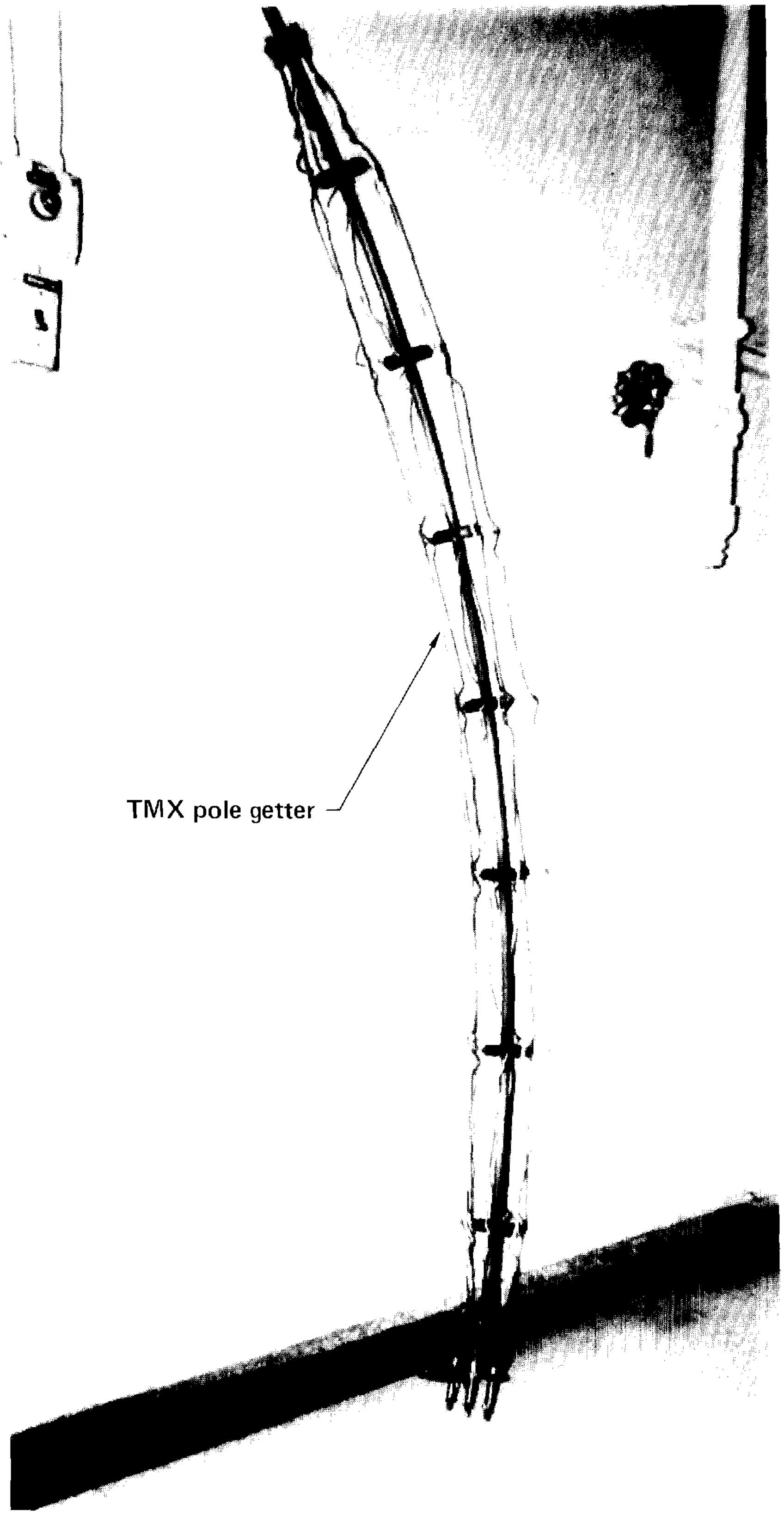



Fig. 3. Pole getters from TMX-U.

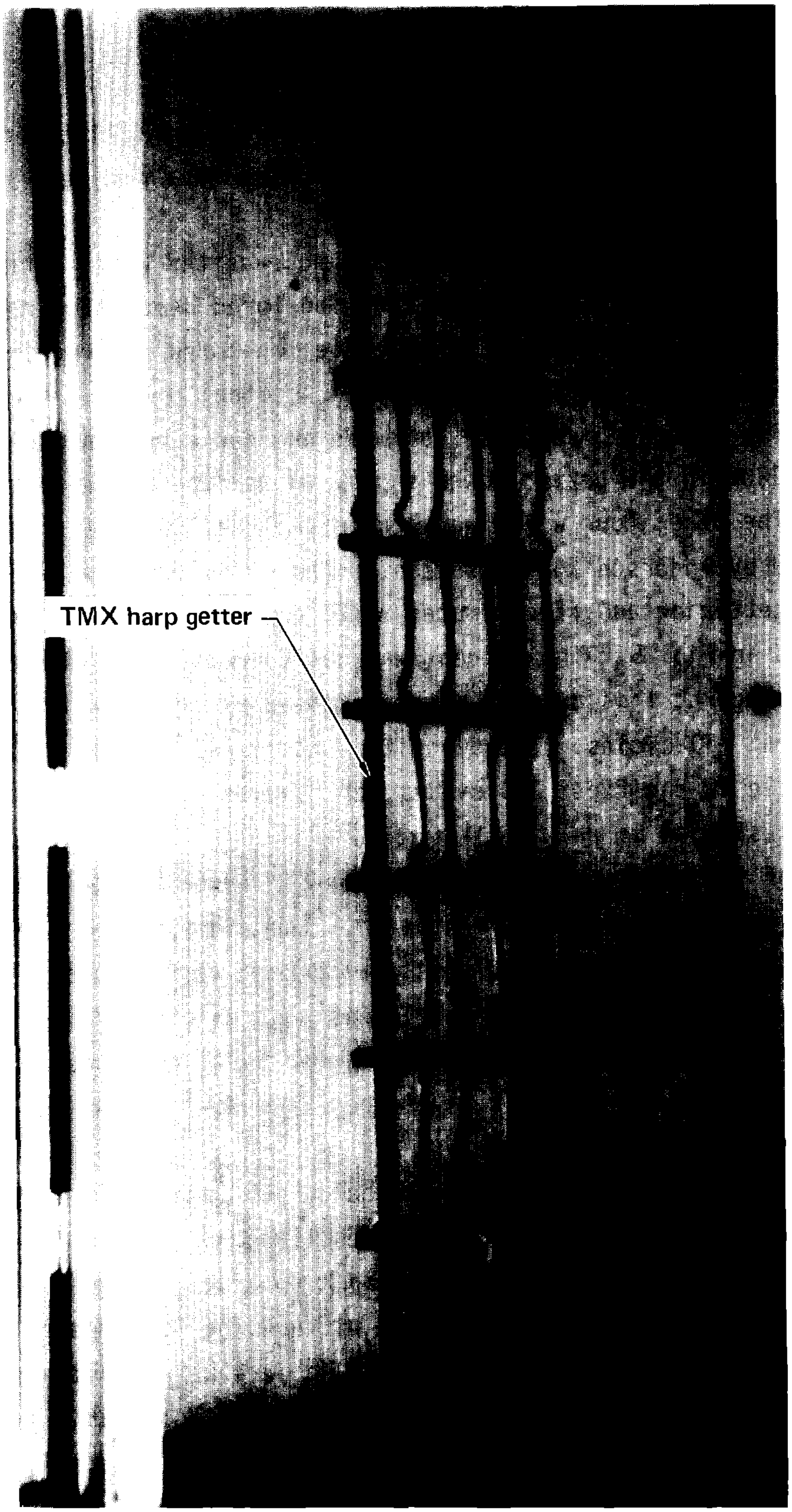


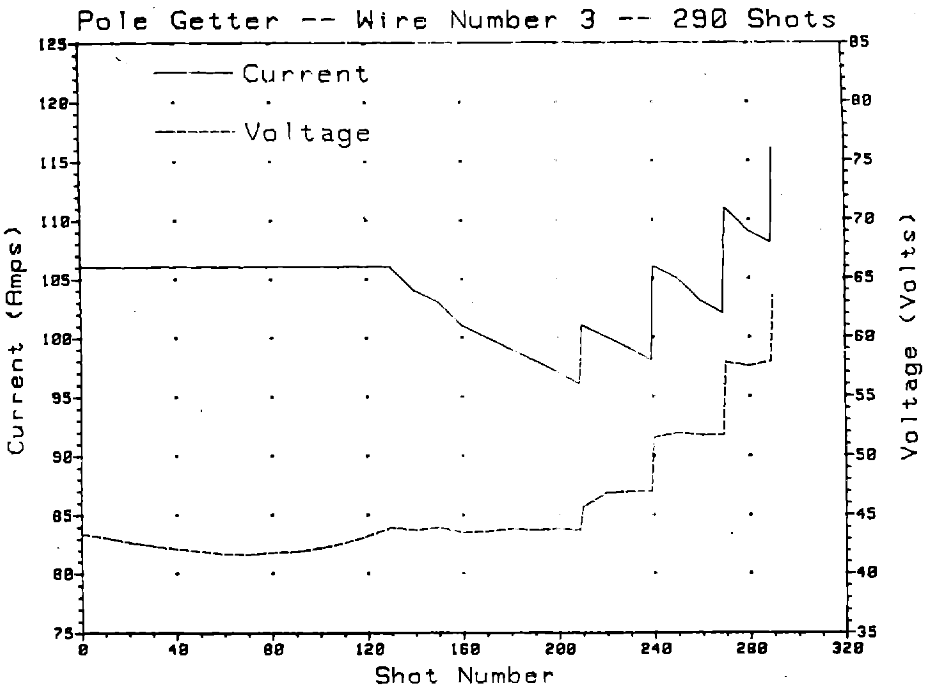

Fig. 4. Harp getters from TMX-U.

supply that was turned on and off by a pair of mechanical timers. All six wires survived the 200 cycles, and the failures that had been seen on TMX-U did not show up here.

We monitored the second set of six pole-getter wires more closely than the first set. The XTM inside the tank monitored deposition rates, and a desktop computer attached to a CAMAC crate monitored and regulated a power supply that had been modified for computer control. Two of these wires were sacrificed to debugging computer software and new electronic circuits that were installed in the getter power supply to monitor the output voltage and current. The other four wires were run using the 106-ampere schedule recommended by Anderson and Finlayson.[2] The wires were cycled with one minute of gettering and five minutes at 20 amperes. We plot data from one of these wires in Fig. 5. The current was not allowed to go above 106 amperes, and the voltage at this current was maintained at or below the initial voltage for the first 210 cycles. The deposition rate rose from an initial 15 Å/cycle to 45 Å/cycle in the first 120 cycles, and showed large jumps each time the current was stepped up.

All of the wires run on the Anderson-Finlayson schedule burned out in the first cycle following a current step. Therefore, three wires were run using the same schedule, except that the current was increased to 1/6 ampere per cycle after 210 cycles. The cycle was also shortened to one minute on and two minutes off. The results of one of these tests are shown in Fig. 6. Although the wires did not last any longer, the deposition rate after 210 cycles was considerably smoother.

The current was maintained at or below 106 amperes for the first 210 shots on the first seven wires tested, resulting in a large peak in the deposition rate as the voltage climbed back up after its initial drop (see Figs. 5 and 6). Four wires were tested without the 106-ampere limit to see how high the current would go if the voltage were held constant and to see how this would effect the deposition rate. One set of these data is plotted in Fig. 7. The current typically rose to about 108 amperes; the deposition peak appeared earlier and was slightly larger than in the previous schedules. The wires only lasted an average of 230 cycles as opposed to 280 cycles with the current limited at 106 amperes.



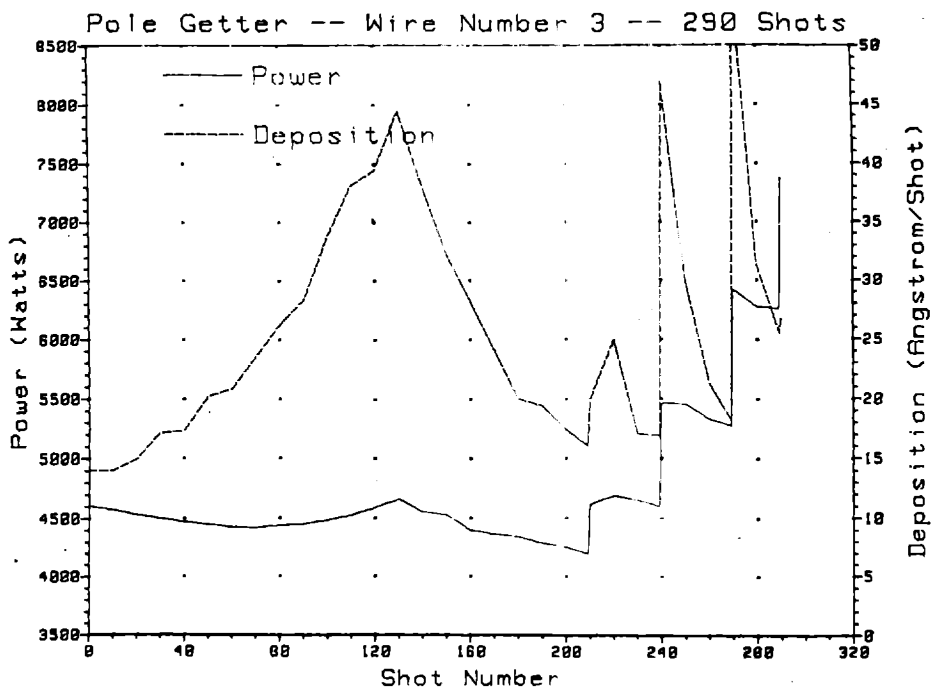




Fig. 5. Anderson-Finlayson current schedule.

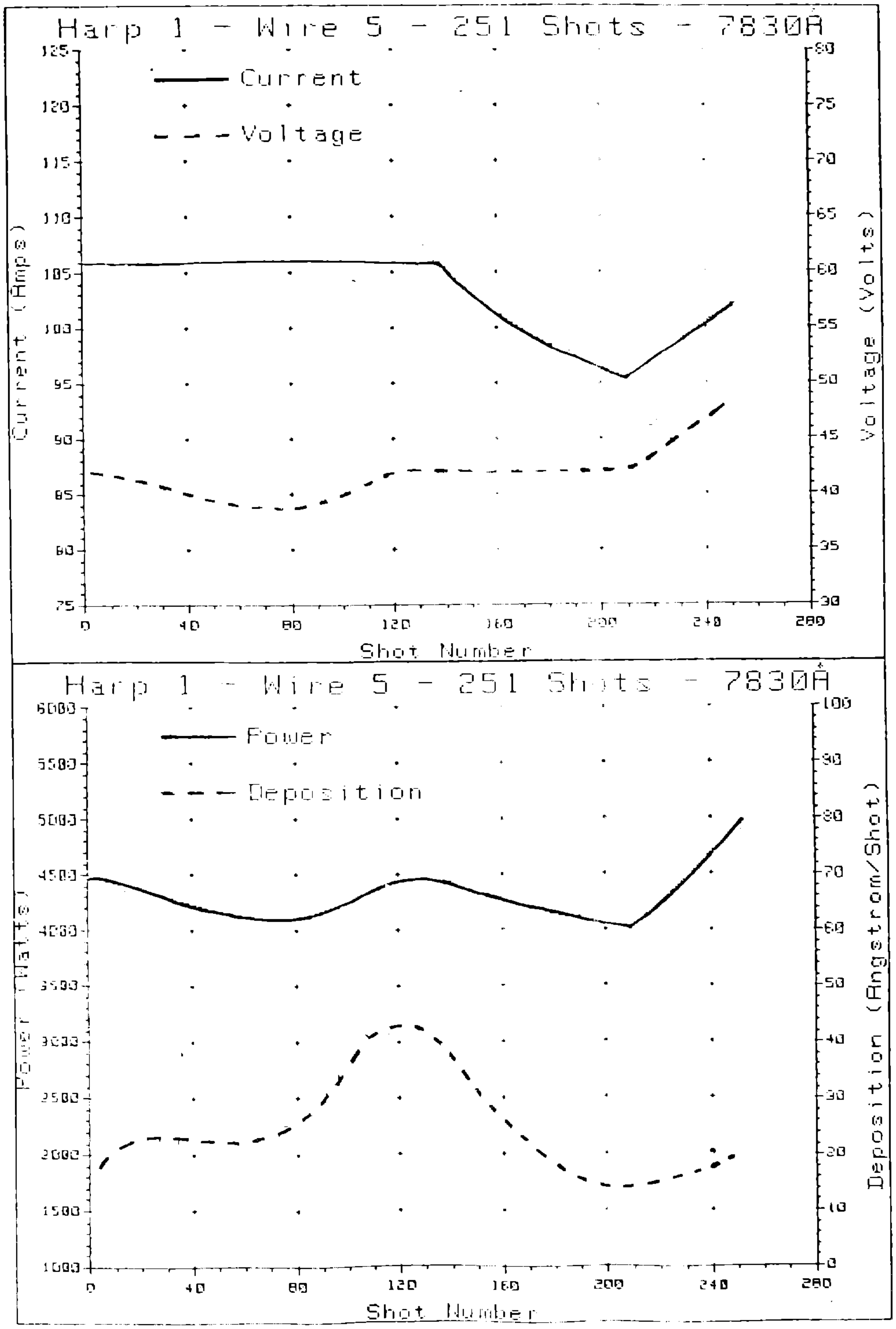



Fig. 6. Anderson-Finlayson current schedule with current ramp after 210 cycles.

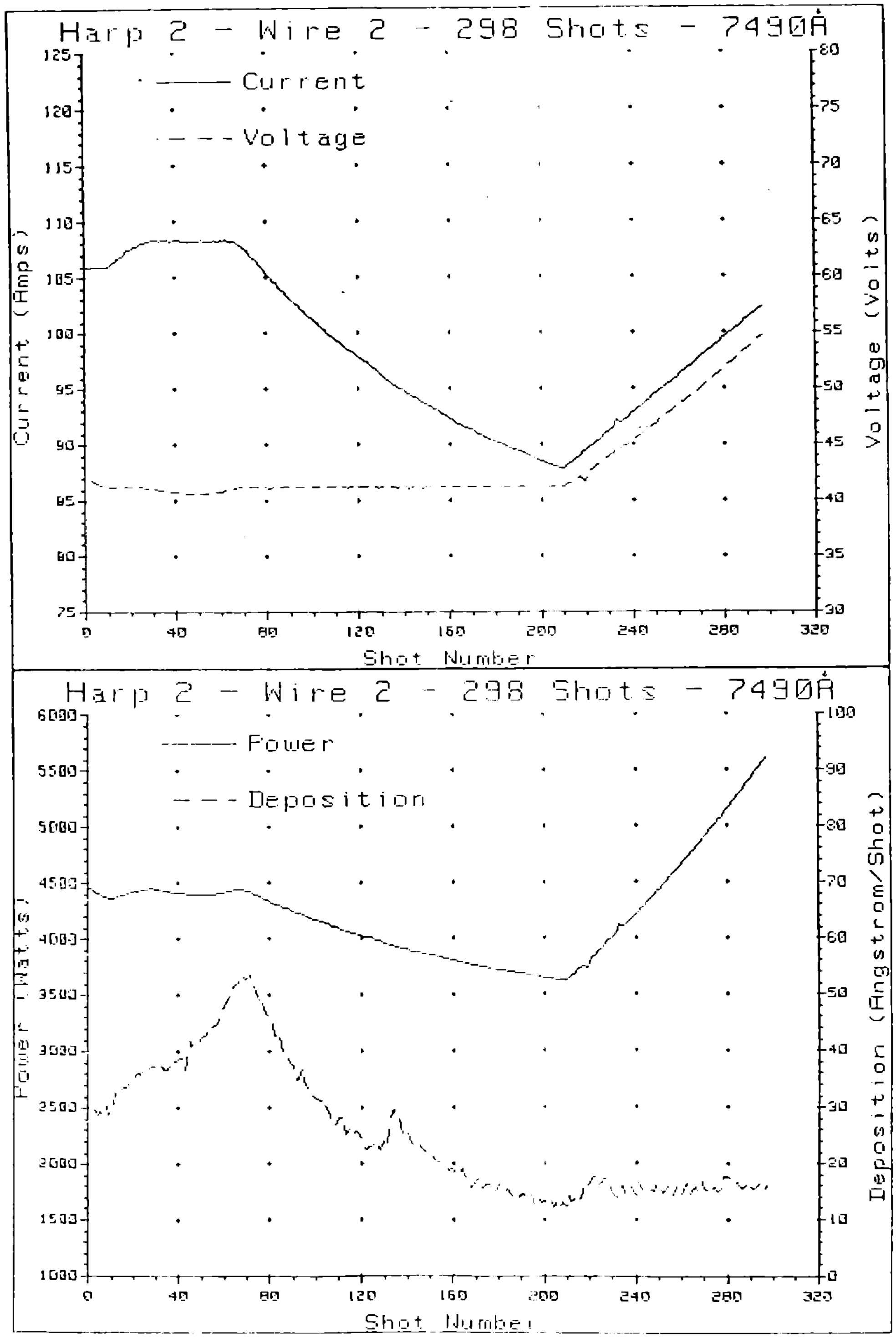



Fig. 7. Constant voltage schedule with current ramp after 210 cycles.

The last schedule attempted to reduce the deposition peak that was occurring early in the Anderson-Finlayson schedule. The voltage was initially allowed to sag with the current held at 106 amperes. When the voltage-monitoring computer detected the voltage drifting back up, it stored and maintained the lowest voltage, until cycle 210 when it started the 1/6-ampere-per-cycle increase. This schedule and the deposition plots are shown in Fig. 8. The peak deposition rate was typically three times the initial deposition rate under the other schedules, and this schedule reduced the peak to about twice the initial rate. The wire lifetime was about 300 cycles.

One wire was run with the deposition monitor in the control loop. The system was programmed to maintain a constant deposition rate of 15 Å per cycle. The wire failed after only 288 shots and it only deposited 4300 Å compared to the 7000 Å typical of other wires. The data for this wire are plotted in Fig. 9. Table 2 is a summary of all the schedules tested, and includes the mass lost by each wire, which was obtained by weighing the wires before and after the tests.

We ran three tests on the wires to help characterize their behavior. The first test measured the temperature of a new wire as a function of current with a 20-mil type S thermocouple spot-welded to the wire. These data appear in Fig. 10. The second test ran a wire for 100 cycles using the current schedule of Fig. 8. The current was then raised to 106 amperes then lowered 2 amperes for each cycle down to 88 amperes, then stepped up, back down, and up again. This schedule is shown in Fig. 11 (cycles 101 to 140) and the results are plotted in Fig. 12.

All of the getter cycles used in these tests and on TMX-U leave the getters on for 60 seconds. The same wire, used to measure getter rates as a function of current, measured deposition rate as a function of getter on-time. For cycles 150 to 170 in Fig. 11, the getter time was decreased, then increased, then decreased again. The results of this test are plotted in Fig. 13. It takes almost 30 seconds to achieve steady deposition and, extrapolating to zero deposition, we see that approximately 15 seconds elapse before the onset of Ti evaporation.

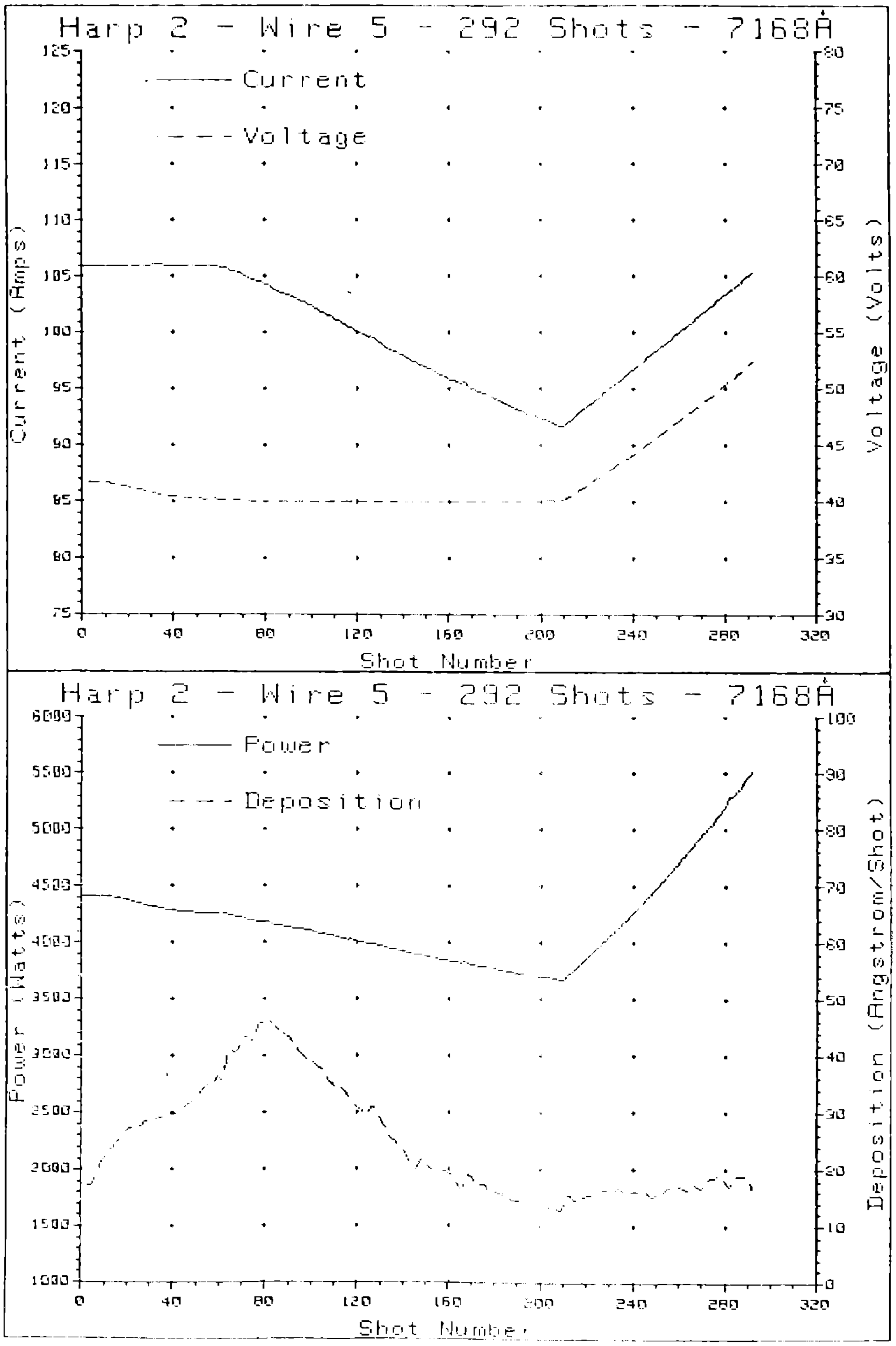



Fig. 8. Voltage sag schedule with current ramp after 210 cycles.

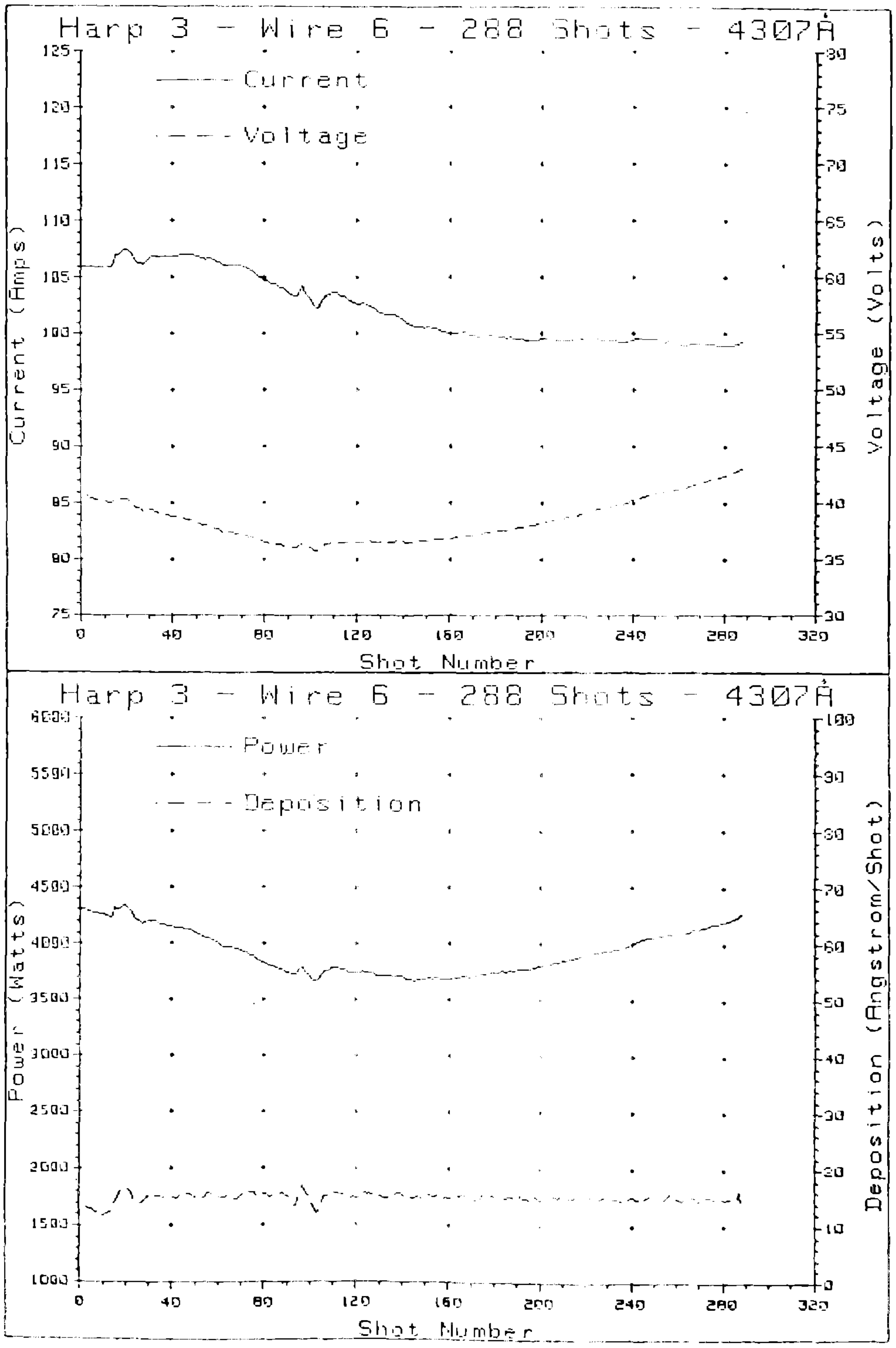



Fig. 9. Constant deposition schedule.

Table 2. Summary of schedules tested.

| Wire | Shots | Mass loss (g) | Deposition (Å) |
|---|---|---|---|
| Anderson-Finlayson | | | |
| 1 | 318 | 26.5 | 6143 |
| 2 | 354 | 26.0 | 6293 |
| 3 | 290 | 28.0 | 8363 |
| 4 | 255 | 18.0 | 5022 |
| average | 304 | 24.6 | 6455 |
| Anderson-Finlayson with ramp | | | |
| 1 | 351 | 30.5 | 8502 |
| 2 | 251 | 28.0 | 7830 |
| 3 | 238 | 26.5 | 6116 |
| average | 280 | 28.3 | 7493 |
| Constant voltage with ramp | | | |
| 1 | 298 | 24.0 | 7490 |
| 2 | 176 | 18.0 | 5750 |
| 3 | 226 | 18.0 | 5703 |
| 4 | 214 | 23.0 | 7003 |
| average | 228 | 20.8 | 6486 |
| Voltage sag with ramp | | | |
| 1 | 326 | 17.5 | 6605 |
| 2 | 292 | 22.0 | 7168 |
| 3 | 334 | 23.0 | 7079 |
| 4 | 264 | 21.0 | 6550 |
| 5 | 258 | 18.0 | 5970 |
| 6 | 314 | 22.0 | 7260 |
| average | 296 | 20.6 | 6773 |
| Constant deposition | | | |
| 1 | 288 | 14.0 | 4307 |

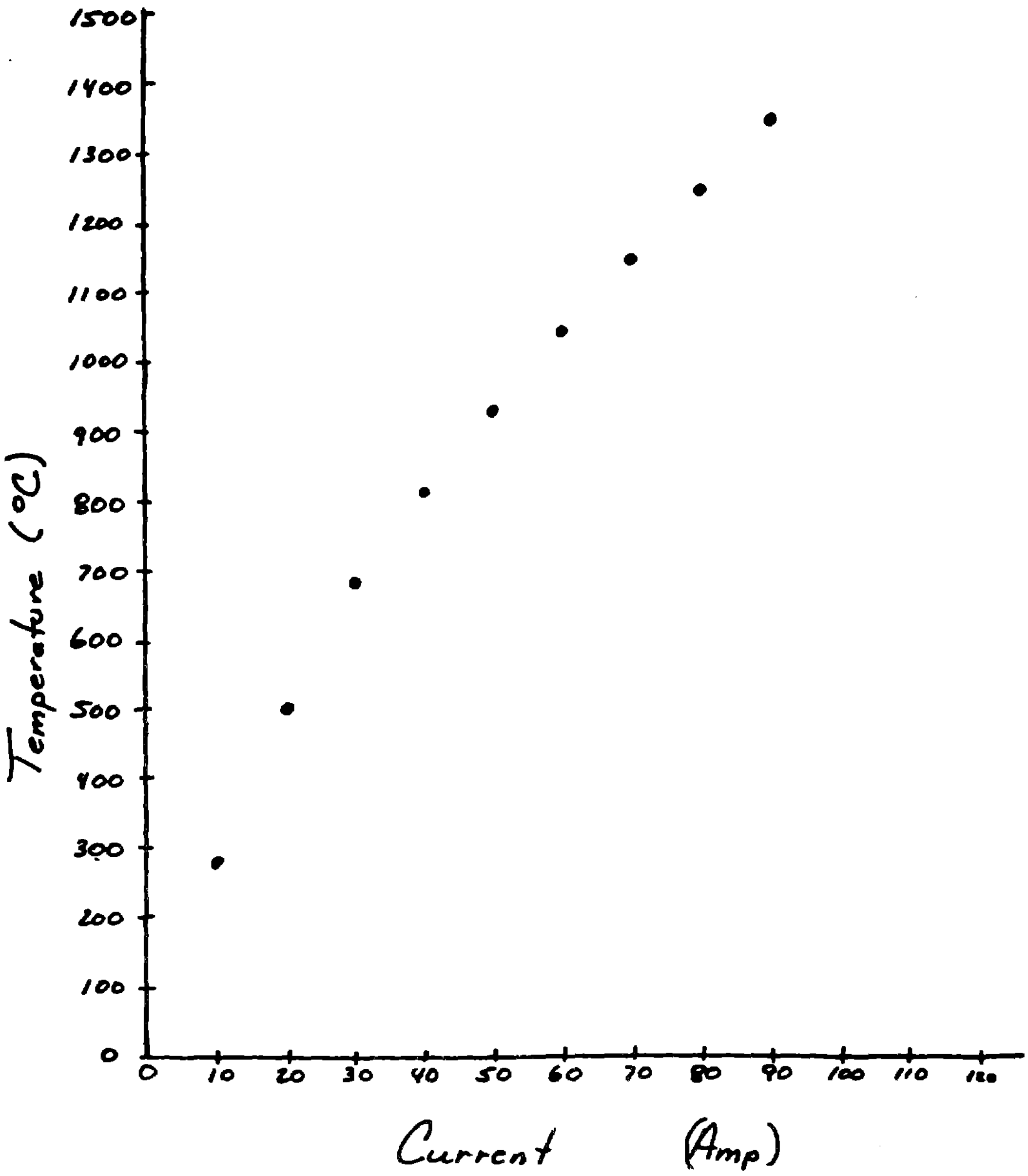



Fig. 10. Temperature vs. getter current.

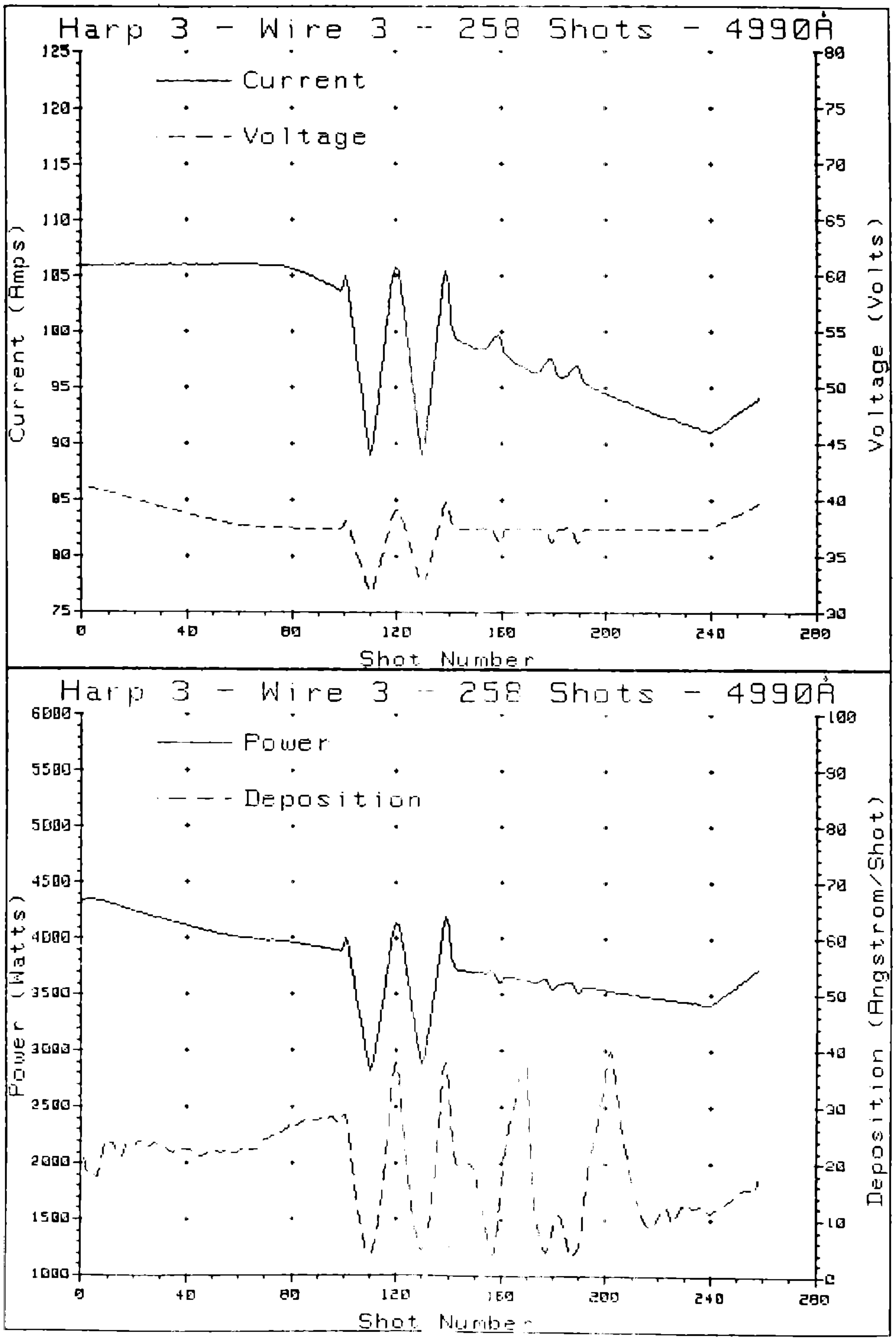



Fig. 11. Current schedule for test wire.

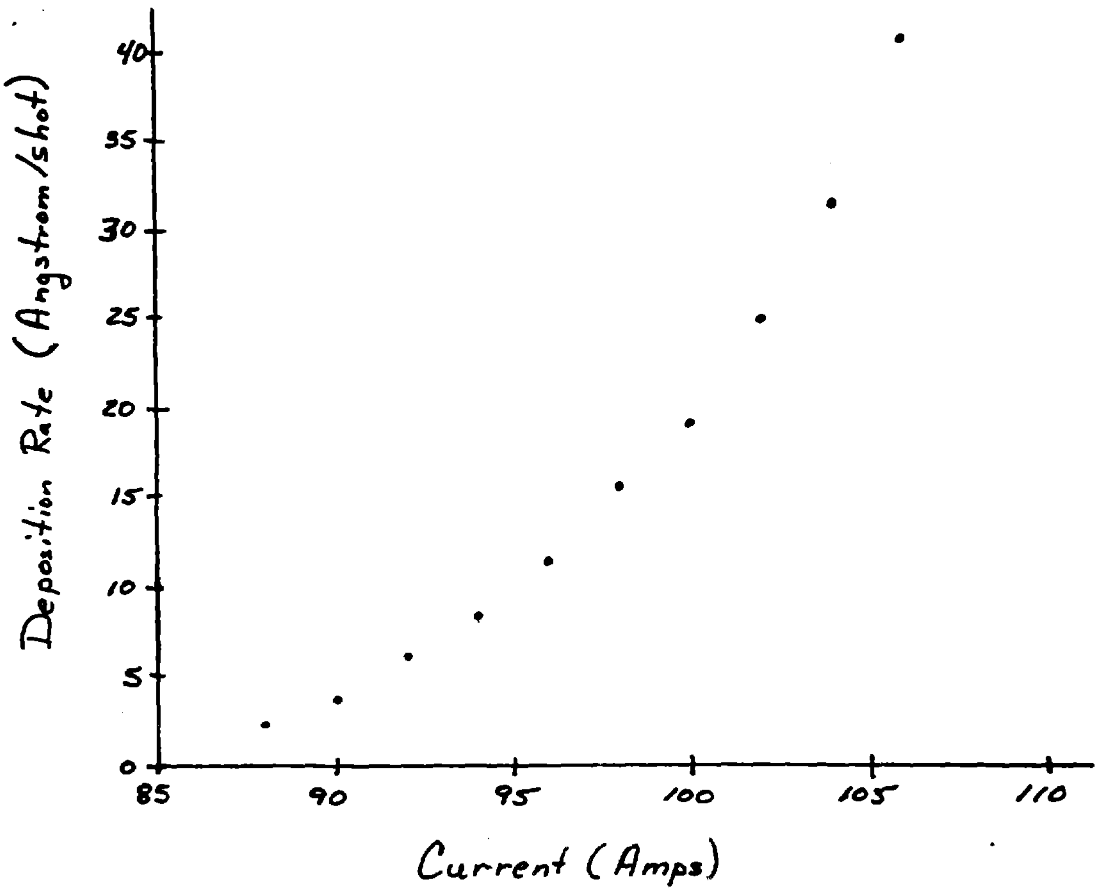



Fig. 12. Deposition rate vs. getter current.

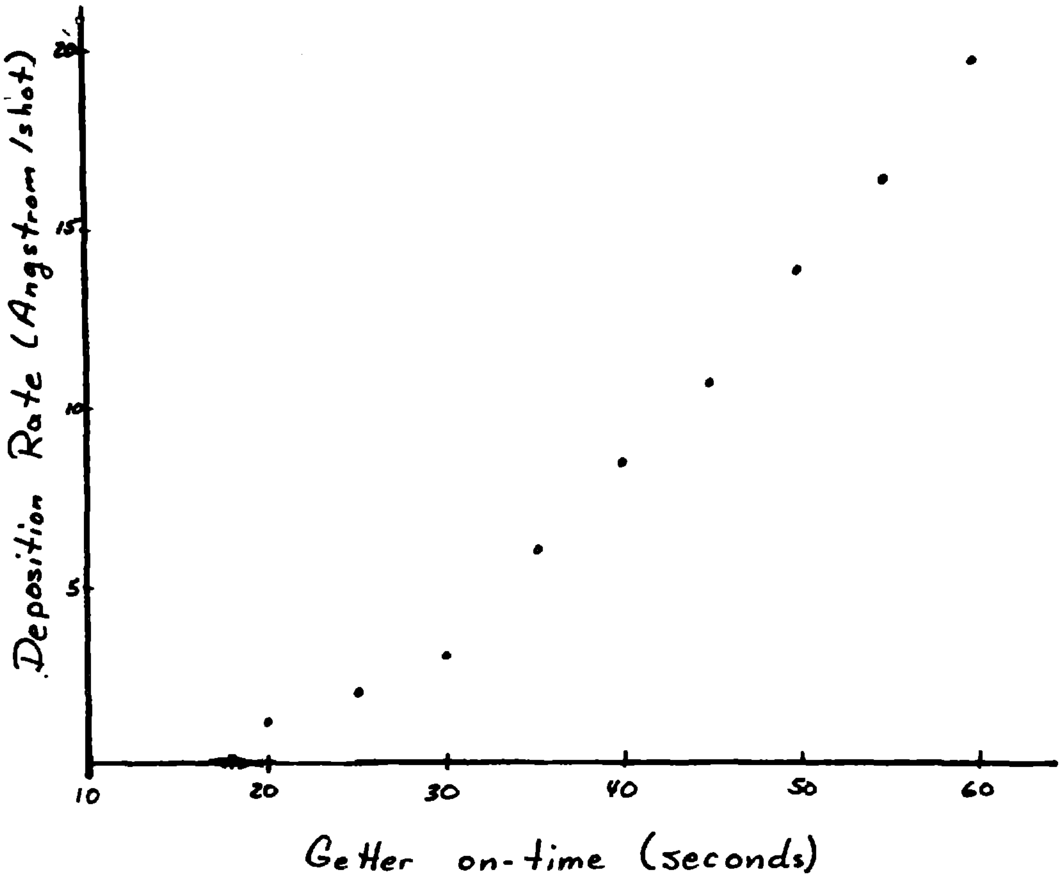



Fig. 13. Deposition rate vs. getter on-time.

## DISCUSSION AND OBSERVATION

Altogether 29 wires were used in these tests. Only one of the wires experienced a premature failure: the wire that had the thermocouple spot-welded to it failed on cycle 99, and fractured at the spot where the thermocouple was welded on. The first six wires were not run to failure, but all except two of the remaining 22 wires failed under tension. These were pulled tightly on the supports, and yielded broken ends separated by 1/4 to 1/2 inch. There was also necking on both ends of the wire at the failure points. The remaining two wires that did not appear to be under tension failed near a bend where the wires were connected to the support posts.

All of the wires were heated for 30 minutes at 80 amperes before gettering with the exception of the first six wires, which were heated for 30 minutes at 60 amperes. The pressure of the vessel rose from a base value of a few times $10^{-6}$ Torr before heating to $10^{-5}$ Torr during heating and climbed to $10^{-4}$ Torr during the first three to five getter cycles. After about 10 cycles, the base pressure was in the upper $10^{-8}$ Torr range and only rose to one or two times $10^{-7}$ Torr during the getter cycle. For a subset of three wires, the vessel was subjected to GDC with the wires in place before being tested. One of the three wires was heated before GDC, and all three were heated after GDC before gettering. GDC did not affect their lifetime, and they all increased the base pressure to the mid $10^{-4}$ Torr range during outgassing, which was 10 times the pressure reached by wires that had not been subjected to GDC.

During these tests no wire failure mode was identified as being due to a material property of the getter wire or GDC. Subsequently, it was found that the high failure rate experienced on TMX-U was due to the inadvertent use of stainless steel support wires instead of tantalum, which is customarily used for these supports. The dominant failure mode was failure under tension as the wires shrank during gettering. The computer-controlled getter schedule shown in Fig. 8 was developed and tested. Since these tests were concluded this schedule has been successfully implemented with computer control on the 162-wire system on TMX-U. The average wire lifetime before failure is 260 to 280 getter cycles.

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3. B. L. Doyle and W. L. Hsu, Sandia National Laboratories, Livermore, CA, memo dated 10/21/83.


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